

PTO: 2007-4702

Japanese Published Unexamined Patent Application (A) No. 13-002864,  
published January 9, 2001; Application Filing No. 11-172550, filed June 18,  
1999; Inventor(s): Shingo Kawano; Assignee: N. O. K. Corporation;  
Japanese Title: EPDM composition

---

## EPDM COMPOSITION

### CLAIM(S)

An EPDM composition prepared by mixing a paraffin group  
processing oil and an ester group plasticizing agent in a peroxide  
crosslinking EPDM.

### DETAILED DESCRIPTION OF THE INVENTION

(0002)

(Prior Art)

EPDM is excellent in heat-resistance and weather-resistance due to its  
structure, and its low-temperature characteristic is excellent even without a  
plasticizer, therefore, is extensively used as a forming material for industrial  
parts.

(0003)

Of these various parts, a gasket and a valve in particular require  
excellent permanent compression warping characteristic, so peroxide cross-

linking group is used for them. But the peroxide cross-linking group has a short induction period for vulcanization, and its starting of vulcanization time is quick. Therefore, for forming parts with complex and thin shapes, the forming material requires fluidity, so 20 phr or more plasticity agent needs to be added to improve the formation defects.

(0004)

In this case, for the plasticity agent, a paraffin group processing oil soluble in EDPM is used. But this plasticity agent is poor in low-temperature characteristic and tends to have a poor recovery rate in a TR test used for evaluating a low-temperature sealing property (See Comparative Example 1). On the other hand, when the plasticity agent is reduced to improve the low-temperature characteristic, the fluidity is reduced and the forming material does not flow at a time of forming, causing the formation defect, such as forming material (See Comparative Example 2).

(0005)

(Problems of the Prior Art to Be Addressed)

The objective of the present invention is to present an EPDM composition excellent for low-temperature characteristic and forming process.

(0006)

(Means to Solve the Problems)

The objective of the present invention is accomplished by EPDM composition, wherein a paraffin group processing oil and an ester group plasticizer are mixed in the peroxide cross-linking EPDM composition.

(0007)

(Embodiment)

As for the peroxide cross-linking EPDM, ethylene- $\alpha$ -olefin-diene copolymer rubber prepared by copolymerizing various diene compounds with ethylene and  $\alpha$ -olefin is used. Crosslinking is generally performed by using an organic peroxide at its ratio about 0.5 – 10 part/weight relative to 100 part/weight of EPDM. The organic peroxide is, for example, dicumyl peroxide, 2,5-dimethyl-2,5-bis (tertiary butyl peroxy) hexane, or 2,5-dimethyl-2,5-bis (tertiary butyl peroxy) hexyne –3.

(0008)

As for the paraffin group process oil and ester group plasticizing agent to be added to the peroxide cross-linking EPDM, their total amount of preferably about 15 part/weight relative to 100 part/weight of EPDM, more preferably, about 20 – 40 part/weight, in case when the flow  $Q_{max}$  value is

0.35 or higher, for example, even in case of using low viscosity EPDM

{ML<sub>1+4</sub> (100°C) : 28}.

(0009)

In the case when the total amount is 20 part/weight, the low-temperature characteristic can be improved if the ester group plasticizing agent is used by 1 part/weight or more, but to produce a sufficient effect, about 3 part/weight is preferably used. As the ester group plasticizing agent is increased in the mixture, the heat-resistance and the permanent compression warping characteristic are decreased. If it exceeds about 15 part/weight, a bleedout appears on the surface of the produce, so about 10 part/weight or less is preferred.

(0010)

As for the ester group plasticizing agents, can be used: butyl of aliphatic group dicarboxylic acid with carbon atoms 4 – 8, such as sebacic acid, azelaic acid, and adipic acid; diethyl such as 2-ethyl butyl, 2-ethyl hexyl, octyl, isooctyl, and isodecyl.

(0011)

To prepare the composition, besides the aforementioned components, a filler or a supplement, such carbon black and silica, oxide or hydroxide of of dimetal, stearic acid, processing oil, are anti-aging agent are mixed if

needed, and kneaded well by a sealed kneader. The cross-linking is performed by press vulcanization at about 150 - 220°C for about 1- 20 minutes and by open vulcanization at about 120 - 200°C for about 0.1 – 20 hours.

(0012)

(Advantage)

By combined use of paraffin group processing oil and of ester group plasticizing agent as the peroxide cross-linking EPDM, the low-temperature characteristic can be improved without costing the forming-workability (fluidity).

(0013)

(Embodiment)

The embodiment examples are explained below.

(0014)

Embodiment Examples 1 – 3

EPDM{ML<sub>1+4</sub>(100°C) : 28}

(EPT14030 by Mitsui Cehmical)	100 part/weight
-------------------------------	-----------------

FEF carbon black	80 part/weight
------------------	----------------

Zinc oxide	5 part/weight
------------	---------------

Stearic acid	1 part/weight
--------------	---------------

2,2,4-trimethyl-1,2-dihydroquinokine	0.5 part/weight
--------------------------------------	-----------------

Zenzimidazole	1 part/weight
Paraffin group processing oil (Diana PW380 by Idemitsu Kosan)	15 part/weight
Dicumyl peroxide (Percumyl D by Nippon Yushi)	3.5 part/weight
Dioctyl sebacate	5 part/weight (embodiment example 1)
Dioctyl azelate	5 part/weight (embodiment example 2)
Dioctyl adipate	5 part/weight (embodiment example 3)

Each component of the above was kneaded in a 10-inch open roll, and the admixture was put through press vulcanization at 180° for 6 minutes.

(0015)

The following measuring was done to the produced vulcanized object.

Also, the forming material fluidity was also measured.

Normal property: Conformance with JIS K-6301

Permanent compression warping: 120°C, 70 hours

Aging test by heating: Normal property change after heating at 120°C for 70 hours.

Low-temperature test: TR10 value (ASTM D-1329) and recovery rate at -30°C.

Forming material fluidity characteristic: 150°C, load 80 Kg, preheating 60 seconds, Φ 1 dies.

See JIS K-7210

(0016)

(Embodiment Example 4)

In the above embodiment example 1, the amount of FEF carbon black was changed to 75 part/weight, the amount of paraffin group processing oil to 19 part/weight, and dioctyl cebacate to 1 part/weight.

(0017)

(Embodiment Example 5)

In the above embodiment example 1, the amount of FEF carbon black was changed to 75 part/weight, the amount of paraffin group processing oil to 17 part/weight, and dioctyl cebacate to 3 part/weight.

(0018)

(Embodiment Example 6)

In the above embodiment example 1, the amount of FEF carbon black was changed to 75 part/weight, the amount of paraffin group processing oil to 10 part/weight, and dioctyl cebacate to 10 part/weight.

(0019)

(Embodiment Example 7)

In the above embodiment example 1, the amount of FEF carbon black was changed to 75 part/weight, the amount of paraffin group processing oil to 5 part/weight, and dioctyl cebacate to 15 part/weight.

(0020)

(Comparative Example 1)

In the above embodiment example 1, the amount of paraffin processing oil was changed to 20 part/weight and dioctyl sebacate was not used.

(0021)

(Comparative Example 2)

In above embodiment example 1, the amount of carbon black was changed to 75 part/weight and dioctyl sebacate was not used.

(0022)

The measurement results in the above embodiment examples and comparative examples are shown in the table below.

Table

Measured items	Embodiment Examples							Comparative Examples	
	1	2	3	4	5	6	7	1	2
Normal property									
Hardness (JIS A)	70	70	69	69	69	68	67	69	69
Wallace hardness (IRHD)	70	69	69	70	69	68	67	69	69
100% modulus (MPa)	6.0	5.8	5.9	5.7	5.8	5.7	5.4	5.8	5.6
Tensile strength (MPa)	18.0	17.7	17.8	18.3	18.7	18.2	18.5	18.3	18.5
Elongation (%)	230	230	230	250	240	250	250	240	240
Permanent compression warping									
120° C, 70 hours (%)	14	15	16	14	14	20	30	15	15
Aging test by heating									
Hardness change (point)	+4	+4	+5	+3	+4	+6	+10	+3	+4
Tensile strength change rate (%)	+1	+2	+7	+4	+1	+8	+20	+6	+5
Elongation change rate (%)	+5	+8	+7	-2	-3	-18	-30	+5	+3
Low temperature test									
TR10 (°C)	-52	-52	-51	-48	-49	-53	-52	-48	-48
-30°C recovery rate (%)	68	67	68	47	57	70	69	45	57
Forming material fluidity									
Flow Qmax (cc/second)	0.35	0.36	0.35	0.35	0.35	0.35	0.35	0.36	0.10



Translations

U. S. Patent and Trademark Office

6/4/07

Akiko Smith